Designation: D 4741 – ~~96~~00

An American National Standard

Standard Test Method for Measuring Viscosity at High Temperature and High Shear Rate by Tapered-Plug Viscometer[1]

This standard is issued under the fixed designation D 4741; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

~~*This standard has been approved for use by agencies of the Department of Defense.*~~

1. Scope

1.1 This test method[2] covers the laboratory determination of the viscosity of oils at 150°C and $1 \times 10^6 s^{-1}$ and at 100°C and

[1] This test method is under the jurisdiction of ASTM Committee D-02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.07 on Flow Properties.

Current edition approved ~~Apr.~~ Dec. 10, ~~1996.~~ 2000. Published ~~June 1996. Originally published as D 4741 – 87. Last previous edition D 4741 – 87 (1996) ε1.~~ February 2001.

$1 \times 10^6 s^{-1}$, using ~~Ravenfield high~~ high shear rate tapered-plug viscometer models BE/C or ~~BS.[3] This test method may readily be adapted to other conditions if required.~~ BS/C.

1.2 Newtonian calibration oils are used to adjust the working gap and for calibration of the apparatus. These calibration oils cover a range from approximately 1.8 to 5.9 ~~cP (mPa.s)~~ mPa-s (cP) at 150°C and 4.2 to 18.9 mPa-s (cP) at 100°C. This test method should not be used for extrapolation to higher viscosities than those of the Newtonian calibration oils used for calibration of the apparatus. If it is so used, the precision statement will no longer apply.

1.3 A non-Newtonian reference oil is used to check that the working conditions are correct. The exact viscosity appropriate to each batch of this oil is established by testing on a number of instruments in different laboratories. The agreed value for this ~~check~~ reference oil may be obtained ~~by reference to~~ from the chairman of the Coordinating European Council (CEC) Surveillance Group for ~~the Ravenfield tapered-plug viscometer method L-36~~ CEC L-36-A90, or ~~to~~ from the distributor.

1.4 Applicability to products other than engine oils has not been determined in preparing this test method.

1.5 This test method uses the ~~centipoise (cP)~~ millipascal seconds, mPa-s, as the unit of viscosity. For information, the equivalent ~~SI~~ cgs unit, ~~the millipascal second (mPa.s)~~ centipoise, cP, is shown in parenthes~~i~~es.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 91 Test Method for Precipitation Number of Lubricating Oils[3]

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[2] This test method is technically identical to that described in CEC L~~-~~36–A90 (under the jurisdiction of the CEC Engine Lubricants Technical Committee) and in ~~Institute of Petroleum method~~ IP 370.

[3] *Annual Book of ASTM Standards*, Vol 05.01.

D 4683 Test Method for Measuring Viscosity at High Shear Rate and High Temperature by Tapered Bearing Simulator[4]
D 5481 Test Method for Measuring Apparent Viscosity at High-Temperature and High-Shear Rate by Multicell Capillary Viscometer[4]
2.2 *Coordinating European Council (CEC) Standard:*[5]
L-36 Test Method for the
L36-A90 The Measurement of Lubricant Dynamic Viscosity under Conditions of High Shear (Ravenfield)
2.3 *Institute of Petroleum (IP) Standard:*[6]
IP- 370 Test Method for the Measurement of Lubricant Dynamic Viscosity u Under Conditions of High Shear u Using the Ravenfield Viscometer

3. Terminology

3.1 *Definitions:*

3.1.1 *apparent viscosity, n*—the determined viscosity obtained by this test method.

3.1.2 *density, n*—the mass per unit volume. In the SI, the unit of density is the kg/m^3, but for practical use, a submultiple is more convenient. The g/cm^3 is 10^{-3} kg/m^3 and is customarily used.

3.1.3 *kinematic viscosity, n*—the ratio of the viscosity to the density of a liquid. It is a measure of the resistance of flow of a liquid under gravity. In the SI, the unit of kinematic viscosity is the metre squared per second; for practical use, a submultiple (millimetre squared per second) is more convenient. The centistokes (cSt) is 1 mm^2/s and is customarily often used.

3.1.4 *Newtonian oil or fluid, n*—an oil or fluid, which at a given temperature, exhibits a constant viscosity at all shear rates or shear stresses.

3.1.5 *non-Newtonian oil or fluid, n*—an oil or fluid which that exhibits a viscosity that varies with changing shear stress or shear rate.

3.1.6 *shear rate, n*—the velocity gradient in fluid flow. The SI unit for shear rate is the reciprocal second (s^{-1}).

3.1.7 *shear stress, n*—the motivating force per area for fluid flow. The area is the area of shear. In the SI, the unit for shear stress is the Pascal (Pa).

3.1.8 *viscosity, n*—the ratio between the applied shear stress and rate of shear. It is sometimes called the coefficient of dynamic viscosity. This coefficient is thus a measure of the resistance to flow of the liquid. In the SI, the unit of viscosity is the pascal second (Pa.-s); for practical use, a submultiple, millipascal second (mPa.-s), is more convenient. The centipoise (cP) is 1 mPa.-s and is customarily commonly used.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *calibration oils, n*—Newtonian oils used to establish the reference framework of viscosity versus torque in this instrument from which is determined the test oil viscosity is determined.

3.2.2 *non-Newtonian check oil, n*—non-Newtonian oil used to check that the gap or distance between the rotor and stator will produce the desired operating shear rate of 1 × 10^6 s^{-1}.

3.2.2.1 *Discussion*—Check oil is an acceptable name for non-Newtonian reference oil.

3.2.3 *test oil, n*—any oil for which apparent viscosity is to be determined.

4. Summary of Test Method

4.1 The lubricant under test fills the annulus between a close-fitting rotor and stator. The rotor and stator have a slight, matching taper to allow adjustment of the gap and hence the shear rate. The rotor is spun at a known speed, and the lubricant viscosity is determined from measurements of the reaction torque by reference to a curve prepared using Newtonian calibration oils.

5. Significance and Use

5.1 Viscosity measured under the conditions of this test method is thought considered to be representative of that at the temperatures and shear rates but not the pressures in the journal bearings of internal combustion engines under operating conditions.

5.2 The relevance of these conditions to the measurement of engine-oil viscosity has been discussed in many publications.[7]

6. Apparatus

6.1 *Ravenfield Tapered PlugTapered-Plug High Shear Rate Viscometer*—Model BE, Model BE/C (single speed) or BS/C (multi-speed). The viscometer uses a rotating tapered plug in a matched stator.

NOTE 1—Model BE/C has a restricted torque range and may not be capable of measuring higher viscosities at 100°C.

[4] *Annual Book of ASTM Standards*, Vol 05.032.
[5] Available from the CEC, Coordination European Council, Madou Plaza, 25th F floor, Place Madou 1, B-1030, Brussels, Belgium.
[6] Available from Institute of Petroleum, 61 New Cavendish St., London WIM 8AR, U.K. 071636 1004.
[7] For a comprehensive review see "The Relationship Between High-Temperature Oil Rheology and Engine Operation," ASTM Data Series Publication 62. Available from ASTM Headquarters.

6.2 *Calibration Weight* *Vacuum Extract Pipe*, (to ensure constant oil level. The extract pipe is supplied with instrument). all current models.

6.3 *Wash Bottle*, fitted with metal tip (supplied Calibration Weight (supplied with instrument).

6.4 *Thermostatically Controlled Heating Bath*, with fluid circulator.6.5 T *For acceptable temperature M control and recovery time, the temperastture difference between the bath and measurement head should be targIeted at 4°C and shall not exceed 8°C. This temperatumre difference is influenced by t,he nature and rate of flow of the circulating fluid; the length and bore of the heating pipes; and the viscosity of the bath fluid.*

NOTE 2—Bath oil with kinematic viscosity not greater than 10 mm²/s at 150°C is recommended.

6.5 A means of measuring temperature is not necessary for current instruments since a precision temperature sensor is now built-in. For older instruments still in the field, a device with a precision not worse than ±0.2°C at 150°C.

6.6 ±0.20°C is necessary.

6.6 The use of an ultrasonic cleaner is recommended.

6.7 The manufacturer offers a package incorporating all the above and including the necessary calibration oils, reference oils, and bath oil.

6.8 *Vacuum Pump*, with suitable liquid trap.

7. Materials

7.1 *Newtonian Calibration Oils*[8],—CEC Reference Oils RL 102-, RL 103, RL 104, RL 105, RL 106, and RL 107.

7.2 *Non-Newtonian Check Reference Oil*[8],—CEC Reference Oil RL 90. 174.

7.3 *Washing Solvent*,—ASTM precipitation naphtha as specified in Test Method D 91 or a solvent of similar volatility.

NOTE 1—**Warning:** Extremely suitable replacement solvent. (**WARNING**—Extremely flammable. Vapors may cause flash fire. See Annex A1.)

7.4 *Flushing Solvent*—While spirit or Stoddard solvent.

8. Sampling

8.1 Test oils that are visually free from haze and particulates need not be filtered before evaluation. A sample must shall be free of particles larger than 3µm. If heavy concentration of smaller particles is still visible after filtration through a filter of pore size 3 µm, a heavy concentration of smaller particles is still visible, 3µm, it is wise at least recommended to reduce their concentration by further filtration. This will reduce the possibility of the particles wedging in the measurement gap and so causing erosion of the rotor/stator or erroneous readings. Do not filter formulated oils through pore sizes below 1 µm because certain lubricant additives could may be removed.

8.2 Used oils may also be tested in these instruments, though no precision statement is available for these materials.

8.2.1 Filter used oils through a suitable filter such as Whatman GF/C fibreglass filter. The process of filtration is greatly accelerated by either warming or applying pressure. Procedures shall be such that all risk of particulate contamination is avoided.

NOTE 3—Suggestions have been made that the process of filtration may itself cause a change of viscosity by the removal of particles. No doubt if there is a very heavy concentration of particles greater than 3 µm, this will be so. It is not expected or intended that this test method will be used for such oils. Evidence to date is that filtration of used oils from normal engines in normal periods of use is acceptable. It is, however, advisable to use pressure filtration rather than vacuum filtration so that volatile components will not be removed. No precision statement is available for used oils.

9. Initial Preparation of Apparatus

9.1 *Insertion of Stator*—For detailed instructions, see Manufacturer's Manual.

9.1.1 Remove the top nylon cover from the stator housing.

9.1.2 Remove the screwed cover.

9.1.3 Place an "O" ring at the bottom of the heating-jacket chamber, making sure it is sitting correctly in the corner of the housing.

9.1.4 Clean the stator thoroughly, preferably using an ultrasonic bath.

9.1.5 Insert the stator into its housing with the thermocouple pocket uppermost and at the left-hand side. Ensure that the stator is sitting squarely in the center of the housing.

9.1.6 Place

9.1 These instructions relate to instruments incorporating a second "O" ring above the stator, positioning it computer, in the corner formed between the stator other words, Models BE/C and the housing.

9.1.7 Place the clamping ring (chamfered end down) on top BS/C. Changes from earlier editions of the "O" ring and replace the screwed cover *as tightly as possible using the fingers only*. Metal-to-metal contact should be achieved both above and below the stator. Failure to do so may result this test method are those given in movement of the stator.

9.1.8 Replace the top nylon cover.

[8] Under the jurisdiction of CEC Engine Lubricants Technical Committee. Ravenfield Designs Limited. are distributors.

9.1.9 Insert the thermocouple into its pocket. Secure the wire in the clamp provided on the top of the instrument base, ensuring that the thermocouple reaches the bottom of the pocket.

9.1.10 Insert the spill-prevention ring.

9.2 *Setting Zero*:

9.2.1 Mechanical zero is determined by the balance of two springs 10.1.5, 10.5.1, 10.5.2, 11.1.2, and is not adjustable.

9.2.2 Transducer zero is set by the manufacturer 11.1.3 and only requires readjustment if the internal mechanism is disassembled.

9.2.3 Electrical zero is set as follows:

9.2.3.1 Remove the rotor coupling from the motor shaft. (Not necessary for Model BS.)

9.2.3.2 Before setting electrical Zero and Span (see 9.3), the apparatus must be switched on (with the motor switched off) for a period use of 30 min to achieve a stable condition. *The heating bath must not be switched on at any time unless a stator is correctly installed vacuum extract pipe to avoid damage to ensure constant oil level (see 6.2).*

9.2 Set up the rotor/stator.

9.2.3.3 Switch apparatus in accordance with the motor on and allow about 10 s for manufacturer's manual. Attach the brake funnel to release and the motor cage to stabilize. (Not necessary for Model BS.)

9.2.3.4 The indicator shows a value varying only one or two digits up or down. The control labelled "Z" may now be used to set side arm, using the indicator, such that it swings symmetrically about 000. rubber sleeve provided.

NOTE 2—Even if the zero appears 4—The funnel has a larger bore than stock funnels in order to increase the ravte of flow of oil samples.

9.3 It is recommended that the instrument ifs NOT mounted, in a fume cupboard since this draws in dirt particles. Local extraction over the heating bath is all that is necessary since the manufacturer's bath is practically sealed.

9.4 When setting up the apparatus, a torque calibration shall be performed following the instructions in the manufacturer's manual.

9.5 The instrument is supplied by the manufacturer with all other functions already calibrated and set up. It is recommended that these other initial settings be accepted until sufficient familiarity is obtained with the rotor inserted into use of the stator (Model BE only).

9.3 *Setting Full-Scale Deflection (Span) apparatus. When it is desired to modify the initial settings, full instructions will be found in the manufacturer's manual.*

9.6 It is advisable to gain access to the list of calibration oils held in the memory of the instrument in order to be familiar with its contents and to check that it is in accordance with the standards actually supplied.

9.7 *Preparation of Apparatus on All Other Occasions*:

9.3.1 Mechanical setting is not possible and is determined by

9.7.1 Turn on the characteristics of the low-temperature-coefficient springs only.

9.3.2 Electrical full-scale deflection is set by applying a known torque and adjusting the digital read-out to match this value. This adjustment shall be carried heating bath.

9.7.2 Flush out after setting electrical zero (see 9.2.3).

9.3.2.1 Detach the top fiberglass cover from measurement chamber using washing solvent.

9.7.3 Refill the instrument, after removing the securing screws.

9.3.2.2 Attach about 800 to 1000 mm of strong cotton thread by a loop to the peg on the machined calibration drum at the top of the swinging frame. About 300 mm from the loop, tie another length of cotton thread about 300 mm long and fasten this to measurement chamber with Reference Oil RL 174.

9.7.4 Leave for not less than half an adjustable support (for example, a ring stand). Attach the calibration weight (supplied) hour for temperature to stabilise.

9.7.4.1 If the remaining free end and adjust the assembly as shown bath does not reach correct temperature in Annex A2.

9.3.2.3 By means of this time, then either extend this period or, preferably, address the control labelled "S" on the panel, set the indicator reading to the applied torque in gram centimetres (calibration weight g × 7 cm drum radius). This adjustment must be carried out with the motor running on Model BE and stationary on Model BS.

9.3.3 After setting full-scale deflection, check the zero setting. It may be necessary to repeat both settings due to a small amount problem of interaction. Both controls retain their setting and regular readjustment why heating is not necessary.

9.4 *Assembly and Installation slow.*

10. Procedure

10.1 *Outline of Rotor Method*:

9.4.1 Fit

10.1.1 The lubricant under test fills the annulus between a close-fitting rotor and stator. The rotor and stator have a gradual matching taper to allow adjustment of the cleaned universal joint gap and hence the shear rate. Spin the rotor at a known speed and determine the lubricant viscosity from measurements of the reaction torque by reference to a line prepared using Newtonian calibration oils.

10.1.2 Use Newtonian calibration oils to adjust the set screw, noting that working gap and for calibration of the n apparatums.

These calibration oils cover a range from approximately 1.8 to 5.9 mPa-s (cP) at 150°C and 4.2 to 18.9 mPa-s (cP) at 100°C. The test method should not be used for extrapolation to higher or lower viscosities than those of the rotor is Newtonian calibration oils used for calibration of the top.

9.4.2 Fit apparatus (see 1.1).

10.1.3 Use a non-Newtonian reference oil to check that the rotor coupling sleeve similarly.

9.4.3 Thoroughly clean working conditions are correct. The agreed value for this assembly, preferably using an ultrasonic bath.

9.4.4 Screw reference oil may be obtained from the Chair of CEC Surveillance Group SL-036 on Method L-36, or from the distributor.[5]

10.1.4 Use six Newtonian calibration oils to prepare a torque versus viscosity calibration. Perform a linear regression to obtain a measure of the fit of the calibration result to a true straight line and of the intercept of torque offset on the zero viscosity line.

10.1.5 The correlation coefficient is defined in Annex A2 and shall be calculated to five decimal places and shall be not less than 0.99970. The torque offset is a useful indication of the quality of a rotor e and stator and its state of running-in. Torque offset may be used as a laboratory quality control parameter.

10.1.6 When a satisfactory correlationg coefficient has been obtained, measure the non-Newtonian reference oil. This oil shall also be used after every three to six test measurements to maintain a continuous check on the correct functioning of the instrument.

10.1.7 The initial measured value for reference oil shall be equal to its value as stated by the motor shaft manufacturer within ±0.04 mPa-s at 150°C and within ±0.06 mPa-s at 100°C. Subsequent measured values for referencke oil shall be equal to its value as stated by the manufacturer within ±0.06 mPa-s, providing it tightly is not in position.

9.4.5 Using the *opposite* direction from the initial deviation from nominal.

10.1.8 If at any clean calibration oil, lubricate point the rotor. Lower check oil measured value falls outside the rotor into acceptable limits, discard all test oil values determined since the lastat successful check oil value and remeasure, following an acceptable check oil determination.

10.1.9 Take readings at the top point of theransition from 149.9°C to 150.0°C or 99.9°C to 100.0°C. This is approximately flush with accomplished automatically in the top Model BS/C and manually in other models. The rate of rise of temperature shall not be faster than 0.1°C in 4 s (0.025°C per s) when operated manually. In automatic operation, the rate of rise may be allowed to increase to 0.07°C per s. A

10.1.10 No *maximum* limit is specified on how long this stage lower rise from 149.9°C to 150.0°C or 99.9°C to 100.0°C may take, but it no further.

9.5 *Setting is suggested that delays of more than 8 or 10 s may make the test method unduly cumbersome to operate. A variation of this period from measurement to measurement will reduce the precision of the test method.*

10.1.11 Take at least two measurements to yield a result. If the difference between successive measurements is greater than 1 %, then take a third or even fourth reading. Such a deviation is nor Rmally indicative of inadequate flushing of a previous sample. An extra flush before taking a measurement may help to obtain accurate results more quickly.

10.2 *Sample Insertion*:

9.5.1 For calibration oil RL 106 calculate the torque to be expected at a shear rate of 1×10^6 s^{-1} using the constants from the calibration certificate supplied with each rotor/stator pair. Make the initial assumption that the active rotor length is equal to "h." (See Annex A3 for the relevant equations.)

9.5.2 With the system at test temperature, carefully adjust the vertical position of the rotor using the handwheel until the top of the rotor is flush with the top of the stator. This may conveniently be done

10.2.1 Insert oils, whether reference fluids or sample fluids, by feeling for a "step" with the end means of an Allen key.

9.5.3 Mount the dial gage funnel mounted on its the side arm and adjust withdrawn by the stop pillar and dial until constant level vacuum pipe to waste.

10.2.2 Fill the gage reads zero.

9.5.4 Lower funnel, then allow to drain into the rotor using the handwheel for eight complete revolutions measurement cell, then refill one or more times, as detailed below.

10.2.3 When inserting an oil of noticeably different viscosity from the dial gage. Raise the ball stop until it previous sample (for example, RL102 following after RL106), use four funnelfuls. Otherwise, use two funnelfuls. One funnelful is supporting the upper system. Turn the handwheel so that the lifting screw approximately 10 mL.

10.2.4 For all repeat measurements, one funnelful is well clear.

9.5.5 Using the washing solvent, flush out the stator several times, turning the rotor gently with the fingers. Apply the solvent to the top of the rotor and remove by suction at the side arm. adequate.

NOTE 3—Solvent should be applied using a wash bottle fitted with a metal tip. (**Warning**—Extremely flammable. Vapors may cause flash fire. See Annex A1.)

9.5.6 Using the following oil flush procedures, introduce calibration oil RL 106 into the measuring system until the rotor is just covered.

NOTE 4—The oil flush procedure, using the oil level pipe and running the motor continuously, is the preferred method for introducing and changing test oil. If quantities 5—The object of test inserting oil are limited, the solvent flush procedure may be used. The solvent flush procedure for a repeat measurement is described in Appendix X2. The precision and bias statements in Section 13 do not apply to the solvent flush procedure.

9.5.7 Mount the suction pipe on the top surface of the instrument with its tip about 4 mm above the top of the stator and about 2 mm away from the rotor drive coupling. The pipe may be bent readily if the height is initially incorrect.

9.5.8 Switch on the suction pump.

9.5.9 Place about 5 to 10 mL of washing solvent into the funnel. This is sufficent to ensure that some is drawn-off by the suction pipe. The use of washing solvent at this stage is to indicated temperature falls. To ensure that the rotor/stator this, it is subjected advisable to a periodic wash try not to minimize buildup of deposits.

9.5.10 Fill the funnel with the first oil sample.

9.5.11 For XBE and BE models, switch on the motor using the motor switch on the front panel. The start-up brake will be heard to operate and release after trap a few seconds. For XBS and BS models, select 3000 r/min, 5 s (RAMP), 99 h (PAUSE), 1 (STEP), then switch bubble below the motor on.

9.5.12 Wait until the oil has fallen to the neck of the funnel. Refill the funnel with funnel.

10.2.5 A minimum temperature drop before making a second portion of the oil sample.

9.5.13 Wait until the oil has fallen to the neck of the funnel.

9.5.14 Switch off the suction pump.

NOTE 5—Since the motor is running all the time, the sample in the measuring section is very thoroughly removed. Two full funnels are sufficient to remove previous oils when viscosities are close. If a high vicosity oil (for example, RL 107) is to be followed by one of low viscosity (for example, RL 102), repeat 9.5.12 to 9.5.13 using a third portion of the oil sample.

9.5.15 Switch off the motor.

9.5.16 Lower the ball stop slowly, gently turning the rotor by hand until a definite resistance to rotation is felt. This increased resistance is usually only felt around part of a revolution. Complete lock-up of the rotor measurement shall be avoided. Record the dial gage reading.

9.5.17 Raise the ball stop for two complete revolutions of the dial gage.

9.5.18 Switch on the motor. The starting brake will be heard to release after a few seconds (Model BE).

9.5.18.1 Alternatively, for Model BS, select 3000 r/min, 5 s (RAMP), 99 h (PAUSE), 1 STEP. Switch motor on.

9.5.19 Observe the torque reading. After a few initial swings, it will be observed first to read an increased torque, as cold oil is drawn in, then drift steadily downwards as the sample heats up.

9.5.20 Allow the motor to run for 30 to 60 s. During this period the temperature will rise due to viscous heating and the torque reading will fall. Record the torque reading at an appropriate temperature during this period. (An appropriate temperature for this purpose is any temperature in the range 145°C to 155°C which fulfills the rate of rise requirements specified in 9.6.3.)

9.5.21 Switch off the motor.

9.5.22 Change the oil for a fresh sample using the oil flush procedure (see 9.5.8 to 9.5.14).

9.5.23 Again introduce calibration oil RL 106, switch on the motor for several seconds, remove the oil by the side arm and recharge.

9.5.24 Repeat operations 9.5.18 to 9.5.20. The same reading should be obtained plus or minus two digits.

9.5.25 Restart the motor and lower the ball stop in small increments, observing the torque. Allow the motor to run for about 5 min at each stage. The rotor should be lowered in this way until the torque calculated at 9.5.1 is achieved at the desired temperature. Halt the lowering process if the required torque is not achieved by the time the dial gage has approached to within 0.03 mm of the figure noted at 9.5.16 and consult the manufacturer's handbook.

NOTE 6—A new or reconditioned rotor/stator assembly requires running in and 9.5.25 must not be rushed. Once a rotor/stator pair has been conditioned in this way, it is possible to advance to a known figure of dial reading and thus achieve the correct torque rapidly.

9.5.26 Having achieved the desired torque reading, it is necessary to recalculate the value using the active length of the rotor to take account of the fact that the active length may be less than "h." The active length is determined using the dial gage reading and the dimensions from the calibration certificate; the necessary equations are given in Annex A3. Fine adjustment results in a position where the torque value is valid for the true active length of the rotor.

9.5.27 Check the calibration with a fresh sample.

9.6 *Temperature Setting* *1°C.*

10.3 *Set Shear Rate*:

9.6.1 Initially, set the bath temperature 1°C to 2°C above the test temperature.

9.6.2 Insert a new sample of oil, that causes the indicated temperature to fall.

9.6.3 Switch on the motor; the temperature will be observed to rise, initially fairly rapidly, then at a lower rate. The target rate of temperature increase

10.3.1 It is 0.1°C in not less than 4 s as the measurement temperature is passed through.

9.6.4 Adjust the bath temperature, initially in 0.5°C increments, then smaller as required, until the condition at 9.6.3 is achieved.

NOTE 7—The rate of shear heating depends upon the viscosity of the test oil, hence some adjustment of the bath temperature will be required when testing oils of widely differing viscosity. With experience, the correct bath temperature may be readily set.

9.7 *Calibration Graph*:

9.7.1 Introduce the next oil using the oil flush procedure (see 9.5.8 necessary to 9.5.14).

9.7.2 If adjust the two determinations of operating gap between the torque agree within 1 %, the average (denoted here as a result) should be used in the plot of torque against viscosity in 9.7.4. If this agreement is not obtained, repeat operation 9.7.1 until any two determinations within 1 % of each other are obtained. Calculate the average of these two determinations. Usually no more than three determinations are required.

9.7.3 Repeat operations 9.7.1 rotor and 9.7.2 with the other five calibration oils.

9.7.4 Plot torque result against viscosity for the six calibration oils, on as large a scale as convenient. This plot must be a straight line, ideally passing through the origin. Alternatively a regression analysis may be used to determine the relationship between torque result and viscosity. Regression analysis shows stator so that correlation coefficients greater than 0.999 are achievable. Any intercept greater than +10 g·cm suggests that metal-to-metal contact may be occurring, in which case cease testing immediately and consult the manufacturer's handbook.

9.8 *Severity Check:* A non-Newtonian check oil is used to eliminate day-to-day variations in the severity of the test and also to minimize interlaboratory variation, thereby improving reproducibility.

9.8.1 Introduce the non-Newtonian check oil into the viscometer on completion of 9.7 and hence determine the torque value for this oil, at a shear rate of shall be $1 \times 10^6 s^{-1}$.

10.3.2 Use Reference Oil RL106, and 150°C, by following operations 9.7.1 and 9.7.2.

9.8.2 Determine adjust to the viscosity from correct torque as instructed in the calibration graph manufacturer's manual.

10.3.3 When, as the temperature passes from 149.9°C to 150.0°C or regression equation. The value should be within 99.9°C to 100.0°C, the accepted limits given for torque indicated agrees with the batch of check oil in use.

9.8.3 Record the dial gage reading.

10. Procedure

10.1 Switch on the heating bath, circulating pump and viscometer electronic unit torque calculated at least 3 h before carrying out any determinations.

10.2 Lower the rotor into current depth indicated by the stator until the frame contacts the ball-stop. Continue winding the handwheel until the lifting mechanism is definitely disengaged.

10.3 Check that the dial gage reading, proceed to 10.4. It is possible that obtained at 9.8.3.

10.4 Carry out the severity check as in 9.8.

10.5 Introduce the test oil as detailed in 9.7.1.

10.6 Remove the sample this may be no longer true after adjustments called for by the side arm and charge with test non-Newtonian reference oil.

10.7 Carry out operations 9.7.1 and 9.7.2 to obtain a torque result, which is the average of two determinations that agree within 1 %.

NOTE 8—When the viscometer 6—This apparent discrepancy is caused by the existence of offsets in the torque measurement system and metallic contact in the rotor and stator.

10.4 *Prepare Calibration Line*:

10.4.1 Use six Newtonian oils to prepare a calibration line of viscosity versus torque. This shall be left non-operational for more than used to prepare a few minutes, it is recommended that linear regression, which shall meet the rotor be raised slightly by means requirement detailed in 10.1.5.

10.4.2 Measure RL102 at least twice to obtain a result as detailed in 10.1.11. Then measure oils RL103 to RL107 in order of ascending viscosity, repeating RL106, and prepare a calibration line as described in 10.1.4 and 10.1.5.

10.5 Use the handwheel. To avoid damage to non-Newtonian reference oil. Measure the equipment, viscosity of the non-Newtonian reference oil.

10.5.1 The value obtained for the reference oil shall meet the requirements detailwed in 10.1.7. If it does not, then mayke adjusbtments as detailed in the manufacturer's manual and repeat 10.4 and 10.5.

10.5.2 Testing shall only proceed when a satisfactory correlation coefficient and a satisfactory value for the heating is switched off. reference oil have been obtained.

11. Calculation

11.1 UsingTest Operation

11.1 *Test Operation*:

11.1.1 Inset a sample oil in accordance with 10.2, note the torque result calculated reading as detailed in 10.7,1.9 and repeat at least once as detailed in 10.1.11.

11.1.2 Calculate the viscosity value from the calibration graph. Alternatively, calculate the viscosity from the linear regression of viscosity on torque obtained with in 10.4.

11.1.3 Repeat the calibration oils. A test report sheet is attached as Appendix X1. measurement of the non-Newtonian reference oil not less often than every six sample results.

12. Report

12.1 Report the apparent viscosity to following information:

12.1.1 The result as the nearest 0.01 cP (mPa.s) high temperature, high shear viscosity at 150°C or 100°C and 1×10^6 s^{-1} $1\times10^6 s^{-1}$ in mPa-s.

12.1.2 The value to two decimal places.

12.1.3 When it is necessary to reduce the number of decimal places in accordance with 12.1.2, this shall be done by rounding to the nearest figure. NOT by truncation. Where the last digit to be rounded is five, the last significant digit shall be rounded up.

13. Test Evaluation

13.1 The evaluation of the test method is performed by observing the correlation coefficient and the reference oil value, and is continuously monitored by use of the reference oil.

14. Precision and Bias

13.1 The following criteria should be used for judging the acceptability of results:

13.1.1

14.1 *Repeatability*—The difference between successive test results, obtained by the same operator, with the same apparatus, under constant operating conditions on identical test material, on successive days, would in the long run, in the normal and correct operation of the test method, exceed the following value only in only one case in twenty:.

2.8 %

1.7 % of the mean

Twelve laboratories were used in a program to obtain at 150°C

1.7 % of the mean at 150°C

1.0 % of the precision statement. Statistical analysis mean at 100°C

1.0 % of the mean at 100°C

14.1.1 This repeatability was obtained using 12 ASTM engine oils in the range established for viscosities from 2.4 2.6 to 4.8 mPa-s. The test series included SAE 0W-30, 5W-20, 5W-30 (4) 10W-30, 10W-40 (2), 15W-40, 20W-50, 4.7 mPa-s at 150°C and 30 grade oils.

13.1.2 from 4.9 to 11.8 mPa-s at 100°C and is independent of viscosity within these ranges.

14.2 *Reproducibility*—The difference between two single and independent results, obtained by different operators working in different laboratories on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the following value only in only one case in twenty:.

5.0 %

2.6 % of the mean at 150°C

2.4 % of the mean at 100°C

14.2.1 This reproducibility was established for viscosities from 2.6 to 4.7 mPa-s at 150°C and from 4.9 to 11.8 mPa-s at 100°C.

14.3 The precision values in 14.1 and 14.2 for 150°C were obtained by statistical examination of interlaboratory results on 12 non-Newtonian test oils tested in 9 laboratories (432 observations in total). The test oil viscosities were between 2.5 mPa-s and 4.8 mPa-s at 150°C and $1\times10^6 s^{-1}$ and viscosity grades were SAE 0W-30, 5W-30, 10W-30, 10W-40, and 15W-50.[9]

14.4 The precision values in 14.1 and 14.2 for 100°C were obtained by statistical examination of interlaboratory results on 9 non-Newtonian test oils in 7 laboratories (126 observations in total). The test oil viscosities were between 4.9 mPa-s and 11.8 mPa-s at 100°C and $1\times10^6 s^{-1}$ and viscosity grades were SAE 0W-10, 5W-30, 15W-40, 20W-40, 20W-50, and 25W-30, 30, and 40.[10]

14.5 *Bias*—No justifiable statement can be made on the—The bias of this test method is less than 0.06 mPa-s. This is guaranteed by the procedure described above, since all determinations are relative requirement to verify periodically the calibration fluid.

13.3 value of a reference oil to within these limits.

14.6 *Relative Bias*—Results from this test method were found, in an by interlaboratory test studyies[10,11] to agree with those from Test Methods D 4683 at both 100°C and D 5481. 150°C. They can be expected to give, on average in the long run, the same results for the same oil.

13.4 A revised procedure has been developed which shows improved precision. This procedure is being tested in an interlaboratory round robin to ensure continued agreement with Test Methods D 4683 and D 5481, and will be balloted as a revision to this test method when available.

14.

15. Keywords

145.1 dynamic viscosity; high temperature; high shear viscosity; high temperature; HTHS; rotational viscometer

[9] Supporting data are available from have been filed at ASTM Headquarters. Request RR:D02-134978.

[10] Supporting data have been filed at ASTM Headquarters. Request RR:D02-1496.

[11] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.07 on Flow Properties.

Current edition approved Dec. 10, 2000. Published February 2001.

ANNEXES

(Mandatory Information)

A1. PRECAUTIONARY STATEMENT— FOR PRECIPITATION NAPHTHA

A1.1 Warning

A1.1.1 Extremely inflammable.
H, harmful if inhaled. Vapors may cause flash fire.
Keep Keep away from heat, sparks, and open flames. Keep container closed.
Use closed, use with adequate ventilation. Avoid build-up of vapors, and eliminate all sources of ignition, especially nonexplosion-proof electrical apparatus and heaters.
A1.1.2 Avoid prolonged breathing of vapor or spray mist.
A1.1.3 Avoid prolonged or repeated skin contact.

A2. TORQUE CALIBRATION

A2.1 See Fig. A2.1 for specified angles.

A3. CALCULATION

A3.1 Let $\dot{\gamma}$ = shear rate, η = dynamic viscosity, and σ = shear stress = $\eta \dot{\gamma}$

A3.2 For any cylindrical viscometer,

$$\text{Shear stress} = (\text{torque})/(\text{surface area} \times \text{radius})$$

$$\text{Hence torque} = \text{shear rate} \times \text{viscosity} \times \text{surface area} \times \text{radius}$$

$$= \eta \dot{\gamma} \times \text{surface area} \times \text{radius}$$

A3.3 Let the rotor

A2. DEFINITION OF CORRELATION COEFFICIENT

A2.1 Definition of Correlation Coefficient

A2.1.1 Correlation coefficients have the following dimensions and been defined in different ways. The correlation coefficient to be totally surrounded used for this test method is defined by the stator (see Fig. A3.1):

A3.4 The area of the rotor curved surface is as follows: formula:

$$\frac{d\pi h\,(2\pi - 4\cos^{-1}(t/d))}{2\pi}$$

Hence,

$$\text{Torque} = \frac{\dot{\gamma}\eta d^2 \pi h\,(2\pi - 4\cos^{-1}(t/d))}{2\pi}$$

or, for a given rotor/stator system

Torque = $\gamma h \eta R$ c

where: R_c = rotor constant

$$= \frac{\pi d^2 (2\pi - 4\cos^{-1} t/d)}{2\pi}$$

$$\text{Torque} = \dot{\gamma}h\eta R_{cr} = \frac{M\sum_{i=1}^{M} x_i y_i - (\sum_{i=1}^{M} x_i)(\sum_{i=1}^{M} y_i)}{\sqrt{[M\sum_{i=1}^{M} x_i^2 - (\sum_{i=1}^{M} x_i)^2][M\sum_{i=1}^{M} y_i^2 - (\sum_{i=1}^{M} y_i)^2]}} \quad \text{(A2.1)}$$

A3.5 The torque will be in N·m when SI units are employed. The instrument manufacturer however uses cgs units and the value of

R_c

where: given in the manufacturer's calibration certificates is in cgs units. (The torque indicator on the instrument is also calibrated in cgs units

M = the number of g·cm) When using the above equation with h in cm, η in cP, $\dot{\gamma}$ in s^{-1} and the value of data points, and

x_i = the observed values of g·cm. This result multiplied by 9.80×10^{-5} will provide the torque in SI units of N·m.

an-dR_c A3.6 Note that the term 'h' two variables.

y_i as given in the manufacturer's certificate, the torque will be in cgs units

This is not included in the rotor constant. This is because the stator is not a simple taper socket but is cut away below the active section. At high-shear rates the rotor always protrudes through the stator; the effective length of the rotor is therefore less than its physical length (see Fig. A3.2).

Thus the effective length h_e of the rotor is given by:

$$h_e = h, \quad \text{when } a < s - h$$
$$= s - a, \quad \text{when } a > s - h$$

The equation for the system is thus:

$$\text{Torque} = \text{constant} \times \dot{\gamma}\eta h_e$$

This equation is used to calculate the torque required with a Newtonian calibration oil of known viscosity to achieve a specified shear rate.

NOTE A3.1—The value for $(s - h)$ is given on the calibration certificate as "depth."

APPENDIXES

(Nonmandatory Information)

X1. Suggested Data Form for Recording Test Results

Date .... Laboratory .... Machine Serial No. ....

Operator ....

Rotor/Stator No. .... Working Depth ....

1. Calibration

Zero .... Gradient ....

Span .... Intercept ....

Correlation Coefficient ....

2. Results

Sample Identification	Torque No. 1	Torque No. 2	Torque 3 if needed	Average Torque	Reported Viscosity

X2. SOLVENT FLUSH PROCEDURE

X2.1 When quantities of test oil are limited, oils may be introduced to the instrument using the following solvent flush procedure as an alternative to the oil flush method in Section 9. The precision and bias statements in Section 13 do not apply to the solvent flush method.

X2.2 Remove the previous oil by way of the side arm.

X2.3 Clean out computer program built into the cell, instruments covered by way of the side arm, with washing solvent (See Note 3).

X2.4 Introduce the new oil by way of the side arm. Check by hand that the rotor is free to rotate. Switch on the motor for about 10 s.

X2.5 Remove the sample by way of the side arm. Charge with a fresh sample of the same oil.

X2.6 Check by hand that the rotor is free to rotate. Switch on the motor. this test method.